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# DESIGN OF STATIC ELASTOMERIC SEALS FOR CRYOGENIC TEMPERATURES

by

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## 1. Introduction

Elastomers have properties which make them ideal for numerous seal applications. Elastomeric O-ring seals enjoy unparalleled and well deserved popularity in almost every area of science and technology. The requirement that a seal remain tight when cycled between room temperature (or higher) and temperatures approaching absolute zero may, however, be considered extraordinary. Very few solids experience more drastic property changes over this temperature interval than do elastomeric polymers. It has often been assumed, therefore, that elastomers are not suitable seal materials for cryogenic temperature applications.

To test the validity of this assumption has been the objective of a continuing study at the National Bureau of Standards Cryogenic Engineering Laboratory for the past three years. Many elastomeric O-ring seals have been tested in flanges of varying design, and highly effective seals for cryogenic applications have resulted. Instead of demonstrating that elastomers are intrinsically unsuitable for low temperature seals, it has been shown that elastomeric O-rings offer simple and reliable solutions for this as well as more conventional sealing problems.

It goes without saying, of course, that the unusual properties of elastomers, and the drastic changes in these properties which occur at low temperatures, must be considered in their use for cryogenic seals. A typical elastomer will change its unstressed linear dimensions by about two percent between room temperature and 76K. It will change from rubbery to stiff or brittle in the neighborhood of 200K; during this "glassy state transition" the modulus of elasticity may change by a factor

* Work supported by Air Force Materials Laboratory, United States Air For



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of 1000 or more. The contraction between the brittle transition temperature ($T_g$) and a cryogenic temperature will normally be much greater than that of the flange material. Thus it is possible for an O-ring to actually shrink away from the flange, leaving a gap instead of a seal. Elastomeric polymers are unique materials which behave in a characteristic manner at room temperatures (where the seal is made) and in an entirely different manner at cryogenic temperatures (where the seal is expected to maintain good performance).

A complete or even typical cataloging of the relation between physical properties and temperature throughout the region of interest would be of great value. This is an almost totally unexplored area, however, and it soon became apparent that we would not be able to base cryogenic seal designs on accurate or well known information. An experimental approach was therefore taken in the design of seals, and an attempt was made to augment this with measurements of properties which were judged to be most significant.

Two essential parts of a mechanical joint are (1) a conformable material which flows under pressure and (2) a mechanical fastener for applying force and holding the joint together. Our objective is to attain the sealing integrity and strength of a weld or reinforced solder joint without losing the convenience of a mechanical connection. Let us examine the two essential parts of a mechanical joint with this objective in mind. We will then be in a position to think critically about cryogenic temperatures and elastomers.

The sealing integrity depends on the conformable material and a sealing pressure which forces it into intimate contact with the surface irregularities. The strength of the joint depends on the mechanical fastener. The sealing pressure may also come from the fastener or, with better design, may come from the pressure of the confined fluid. Many good designs make an initial seal with the help of force from the fastener, after which the seal is energized by the pressure of the enclosed fluid. An elastomer O-ring normally operates in this manner, as shown in figure 1. This extremely simple and effective action is the key to the unmatched success of elastomeric O-ring seals.

Another popular way to use the fluid pressure to energize a seal is illustrated in figure 2. Here the initial seal is obtained by light loading which flexes a metal body. After this the fluid pressure acting

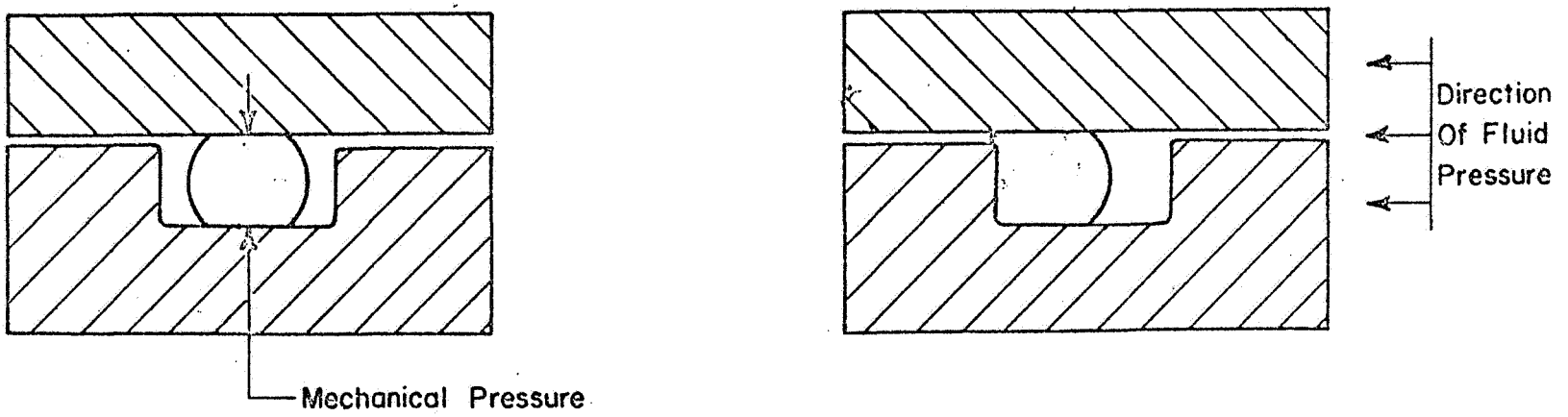



FIG 1. Elastomer O-Ring, Nominal Operatio

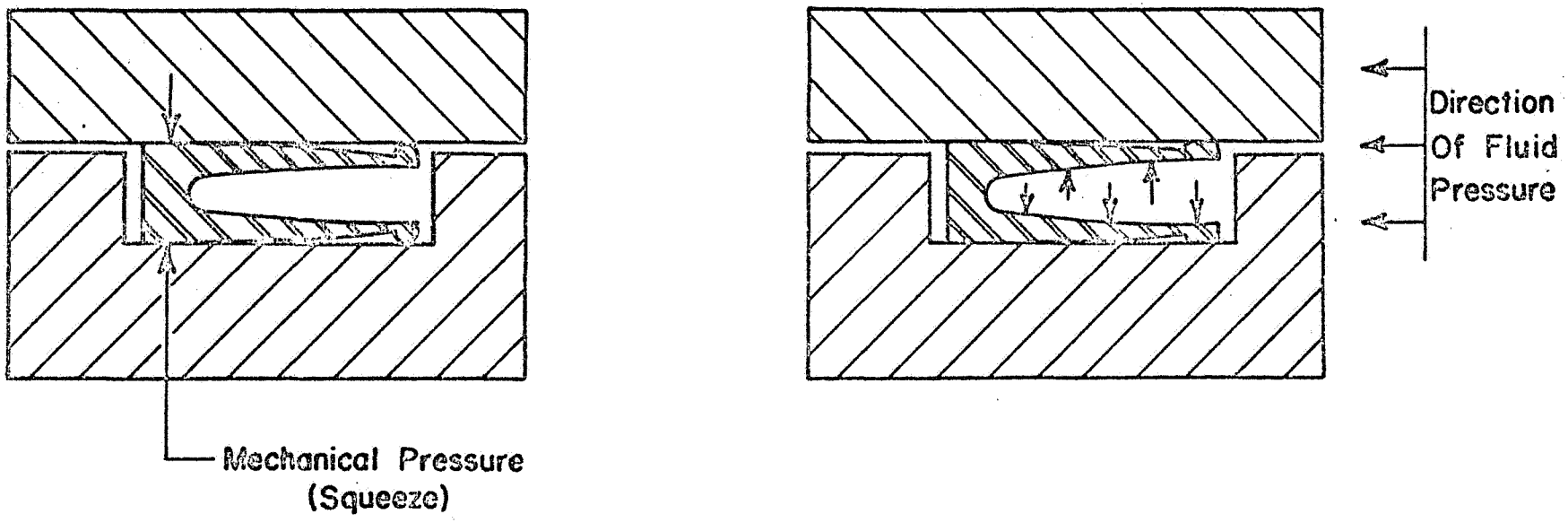



FIG 2. Pressure Actuated Metal Body Seal

on the area inside the body results in a force which is concentrated on the sealing edges. The sealing edges or lips are usually coated with a soft metal or plastic. This type of seal is particularly effective in small sizes, but it is expensive and requires very accurate flange surfaces. It's main advantage is light flange loading and the ability to follow axial movement without drastic reduction in sealing force. Its weakness lies in the small amount of material which flows to make the seal; this makes it susceptible to minute surface imperfections, as well as to unseating which may result from relative motion between the sealing surfaces. Such motion may be caused, for example, by different contraction rates between the flanges and the seal body.

When considering elastomers for cryogenic static seals we are thinking primarily of the first of the two essential parts of a mechanical joint, i.e., the conformable material. Without worrying about efficient flange or fastener design, the first part of our investigation consisted of experiments designed to see what the elastomer itself would do when used as a sealing medium and then cooled below its brittle point. The seal will unquestionably be good at room temperature. What is questionable concerns behavior when the seal is cooled from room temperature to the cryogenic region. Specifically, we should like to know:

(1) If the seal leaks, at what temperature and pressure does the leak begin?

(2) What physical changes cause the seal to leak?

(3) Which of the available elastomer compounds perform best?

## 2. O-Ring Seal Experiments

It was easily shown that elastomeric O-rings can make excellent seals at cryogenic temperatures. The first successful seals used a "brute force" approach, as shown in figure 3. A tongue and groove type of flange was used, giving the closely confined O-ring an 80 to 90 percent linear compression followed by about five percent volume compression. High flange loading was necessary (on the order of 10,000 lb/in of circumference) but the seals were helium leak detector tight at 1000 psig and 20K. When vibrated on a shaker table at 70K they remained tight[1]. The better compounds, moreover, emerged from this drastic treatment with no visible sign of material failure, although some compression set was usually observed.

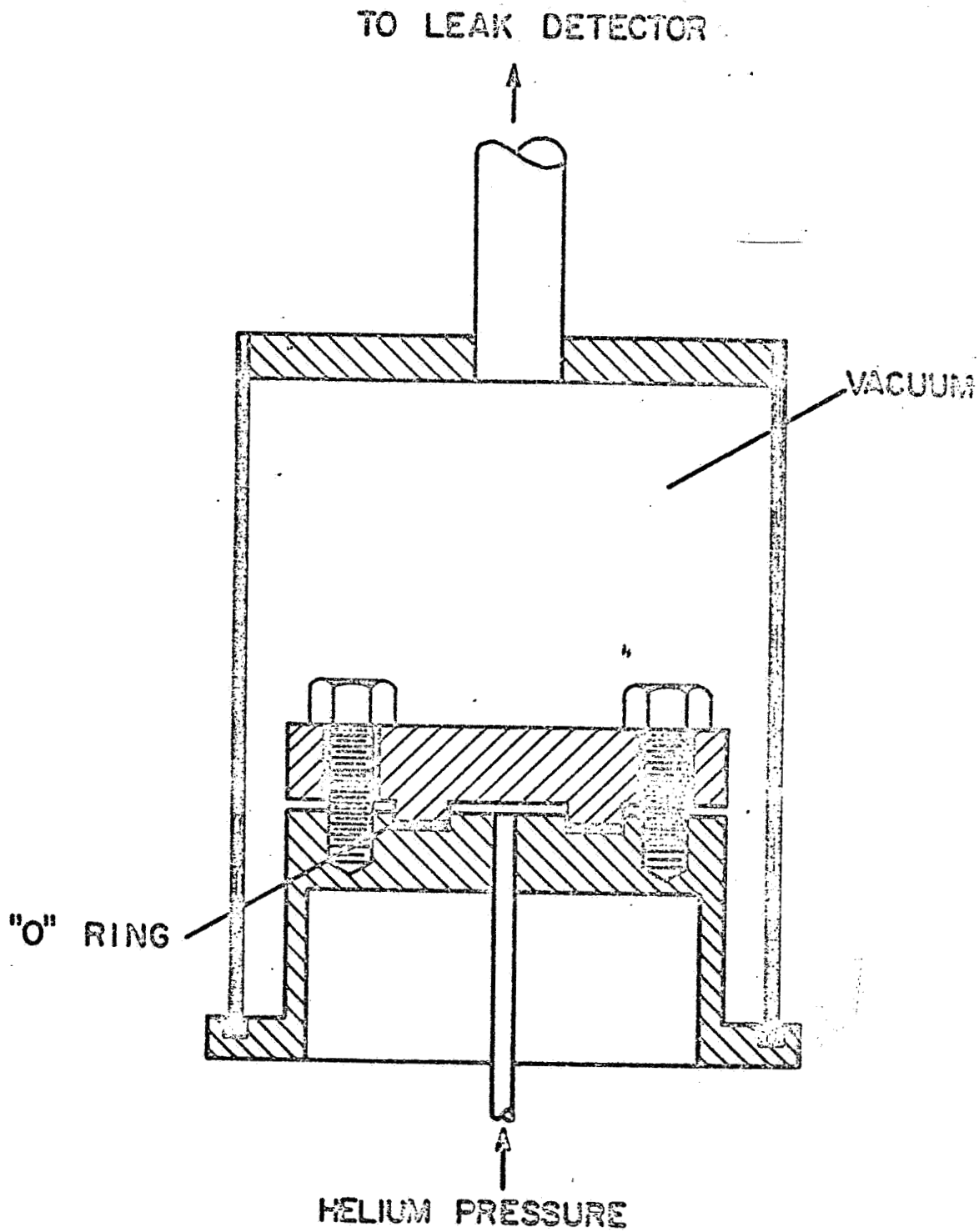



FIG 3. High Compression Torque and Groove

The next step was an effort to reduce flange loading and simplify flange design by eliminating the tongue and groove[2], as shown in figure 4. This also resulted in successful seals and these could be maintained with lower flange loads than had been used in the tongue and groove design. Further reduction in flange loading was achieved by using an O-ring of smaller cross section. When the 0.139 inch cross section O-rings were replaced with O-rings having cross section diameters of 0.070 inches, good seals were obtained with flange loading around 6000 lb/in of seal circumference. These tests were performed with stainless steel flanges 3/8 to 1/2 inch thick which flexed enough to provide a degree of incidental spring loading on the compressed O-ring, a feature which helped to maintain the sealing force. This feature was further exploited by the flange design shown in figure 5.

Here an O-ring of 0.070 inch cross section was lightly stretched around a retaining sleeve which snugly fit the I.D. of the flanges. The flanges were dimensioned to flex appreciably under the bolt load required to compress the O-ring. With this arrangement for positioning and partly confining the O-ring, it was possible to obtain reliable seals with flange loads as low as 2000 lb/in of seal circumference.

These tests demonstrated principles to be employed in the use of elastomeric O-rings as simple crush gaskets for seals at cryogenic temperatures:

(1) Compress as small a cross section as is practical.

(2) Compress to the minimum thickness which does not result in material failure.

(3) Spring load the compressed elastomer in some effective way.

At the same time it was recognized that the tests demonstrated principles only, that the flange designs used in the testing were not optimum and in some cases not even practical, and that no discriminating comparison of the performance of various elastomers had been made. We proceeded next to make a systematic comparison of the performance of various elastomers, and then to seek the most practical and efficient flange designs for use of these materials.

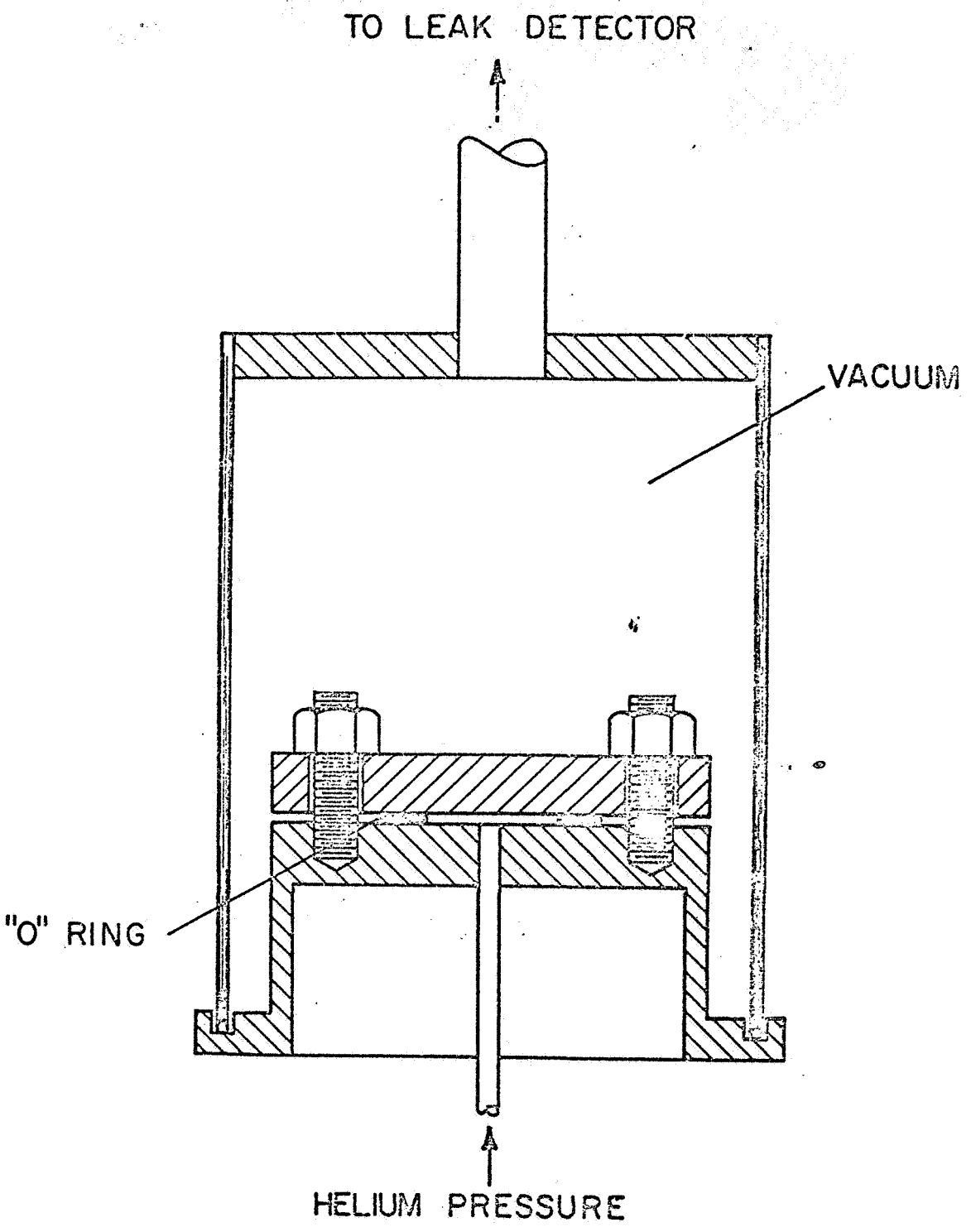



FIG 4. Flat Flange Test Fixture

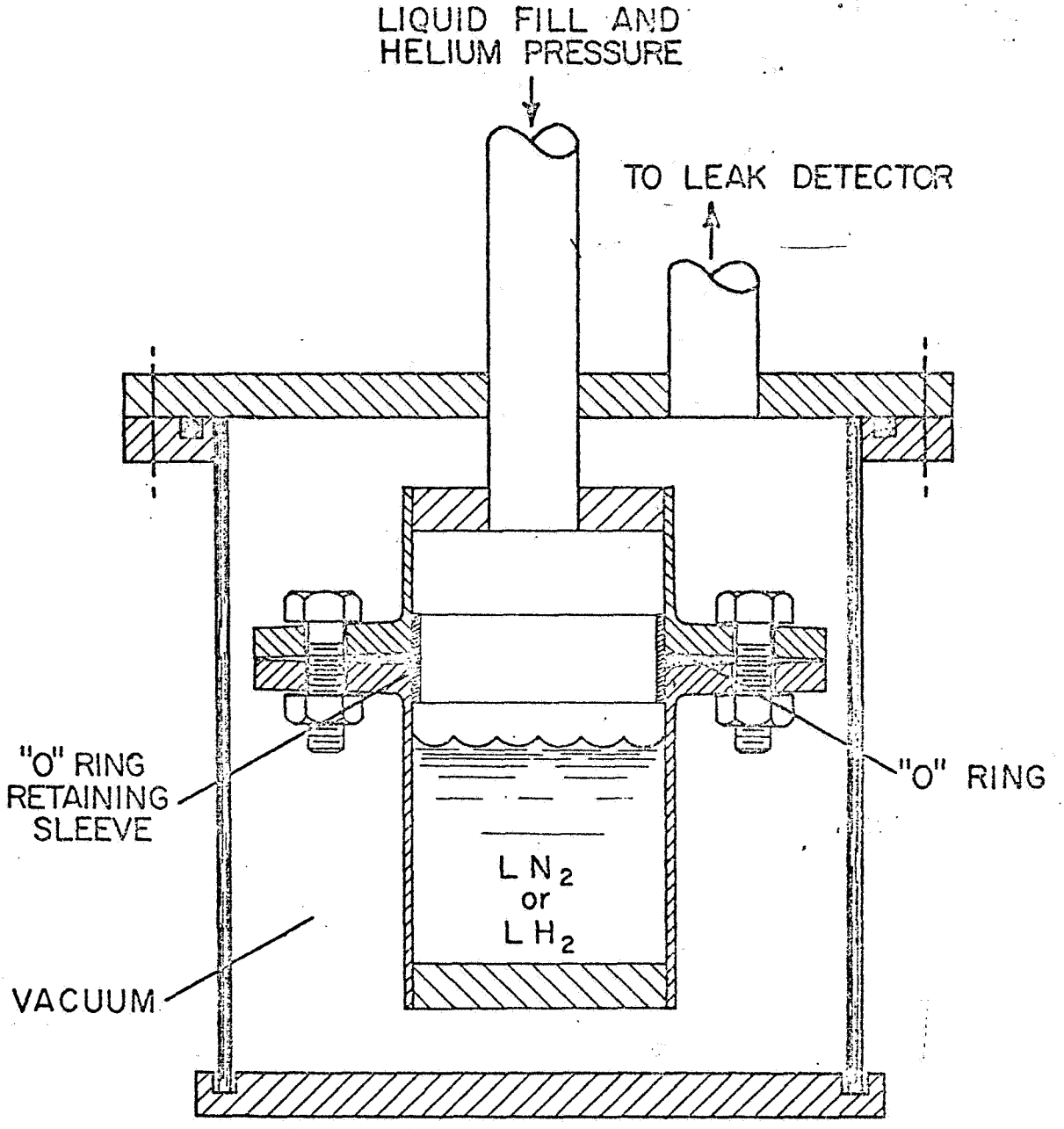



FIG 5. Flat Flange and Retaining Sleeve

## 3. Force Decay Seal Tester

An apparatus was designed to critically compare the performance of various elastomers when highly compressed at room temperature, then cooled to cryogenic temperatures while maintaining a seal. Conditions of the test were such that most of the seals would leak, with the temperature at which a leak occurred serving as an index of merit. The apparatus is shown in figure 6. Compressive force was applied through washer type strain gauges placed in a bellows filled with liquid nitrogen during cooldown.

Initial compressed thickness of the O-ring was maintained as constant as possible by making critical parts of the apparatus of invar and placing invar pillars between the loading bolts. This maintained a constant separation between the main flanges and eliminated most of the spring loading, so that we were able to obtain a record of the force which the compressed elastomer exerted against the flanges during an initial relaxation period before cooldown and during cooldown to 76K. Thus parameters obtained for use in comparing the various elastomers were:

(1) The temperature and pressure at which a leak occurred.

(2) The force at which a leak occurred.

(3) The force decay during initial relaxation.

(4) The force lost during cooldown.

Data were plotted with the help of an x, y recorder, giving us graphs such as those shown in figure 7. Three successive cooldowns were made for each material which showed promise, with seals separating high vacuum from helium pressures of 100 to 1000 psig.

Detailed results of this work are given elsewhere[3]. For our present purpose we can summarize by saying that about 50 different samples divided into 10 groups representing all of the basic elastomeric polymers were tested with this apparatus, giving about 25 graphs similar to those we have seen. From a study of this information we were able to reach several conclusions regarding the use of elastomers for cryogenic seals. Among these were the following:

(1) Even with constant flange separation and a minimum amount of spring loading, some elastomers are capable of maintaining high sealing forces when cooled far below their brittle transition temperatures.

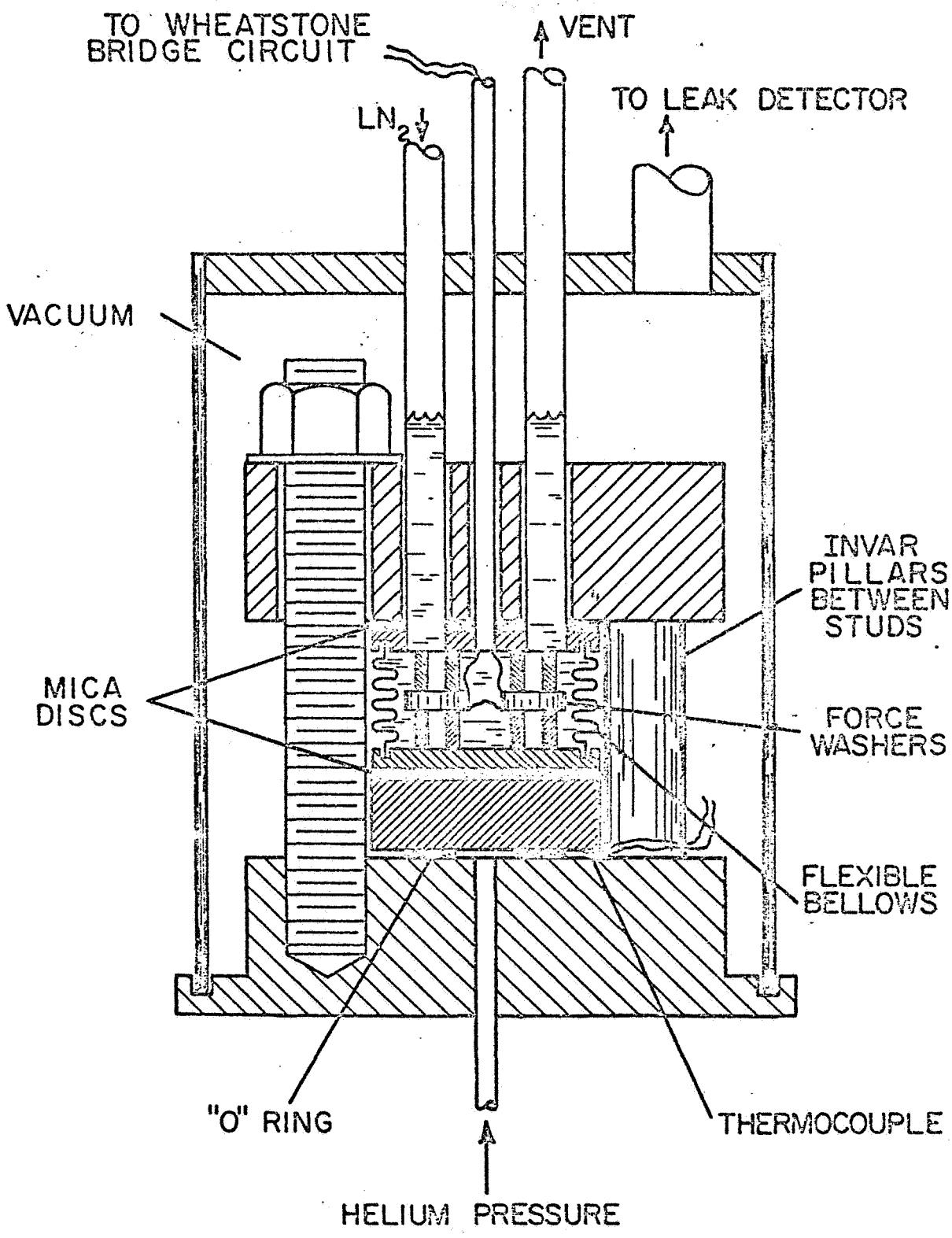



FIG 6. Force Decay Test Fixture

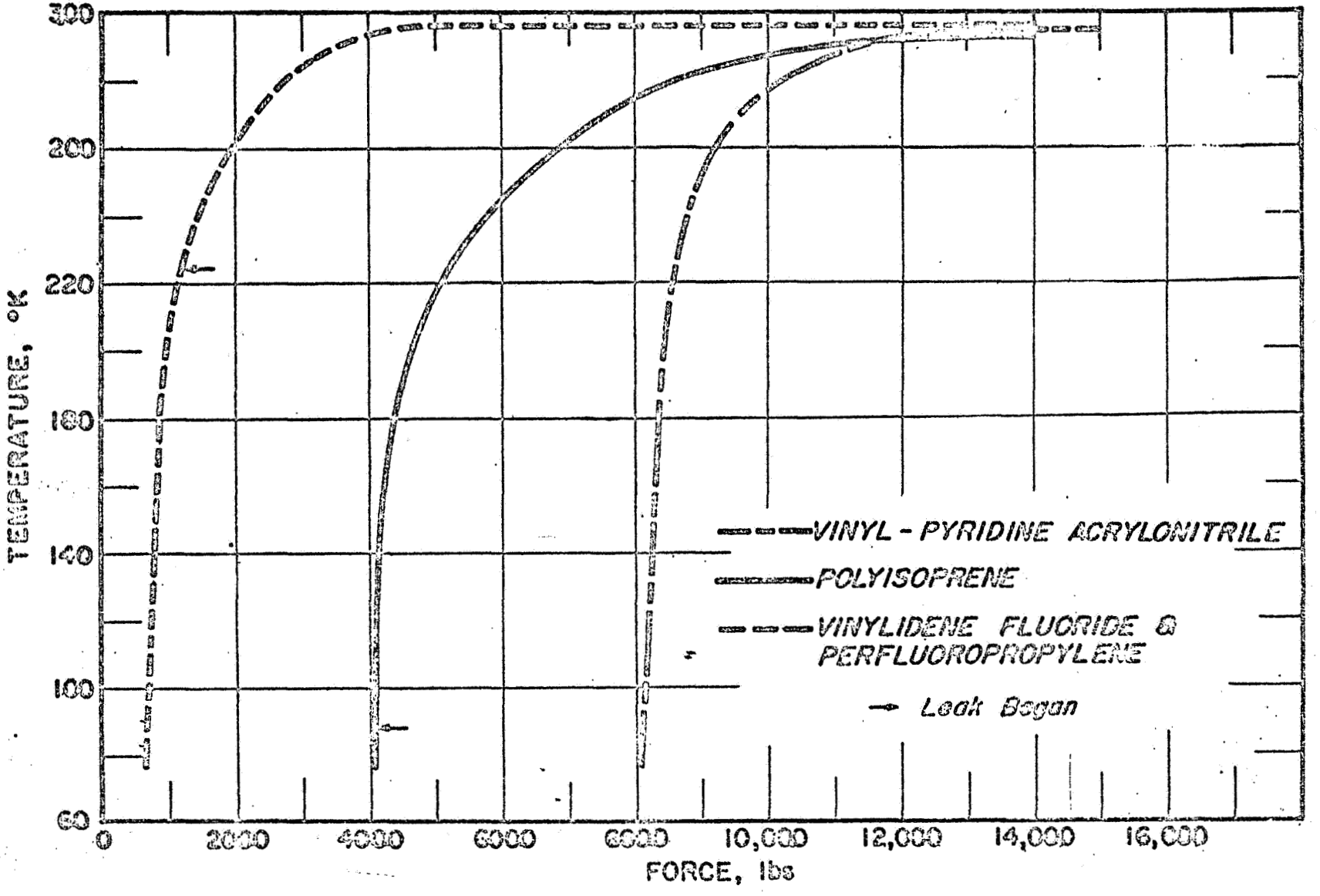



Figure 7 Force decay during cooldown

(2) With high compression and small final thickness most of the force decay occurs during an initial relaxation period of about six hours at room temperature. The amount of this initial relaxation is a fair indication of the effectiveness of the material for cryogenic seals.

(3) Best seals were obtained with compounds of natural rubber, polyisoprene, polybutadiene, vinyl pyridine-acrylonitrile, neoprene, and butadiene-acrylonitrile. Several others, including one experimental silicone, also showed promise.

(4) Many of the seals leaked while the elastomer was still maintaining considerable net force (three to five thousand psi) against the flange surfaces. This apparently was the minimum sealing force which had to be maintained. The reason for this was not obvious, and led to an investigation of sealing and unseating mechanisms.

## 4. Seal Interface Studies

Since the contraction rate of the brittle elastomer is several times as high as that of the confining flanges, temperature changes which occur below $T_g$ will introduce thermal strains parallel to the flange surfaces, and may even cause lateral motion or sliding along the interfaces. Relative motion at the interfaces will result in "unseating" of the seal unless there is plastic flow or deformation to prevent this. The seals which leaked while the net sealing pressure was still high were probably operating in this way. A "constant force" experiment was designed to study this problem and to find ways of reducing the required sealing force.

The central part of the constant force apparatus is shown in figure 8. Force was applied to the flanges at what would normally be the bolt circle, but instead of using bolts a hydraulically loaded testing machine was used to maintain a constant compressive force during cool-down. A specially designed dewar, not shown in the figure, surrounded the apparatus for cooling by slow addition of liquid nitrogen. The O-ring was positioned around a machined step on the bottom flange; the top flange had a circular recess which fit the step with 0.002 inches diametral clearance. Thus the O-ring was confined at its I.D. and forced to extrude outward when compressed.

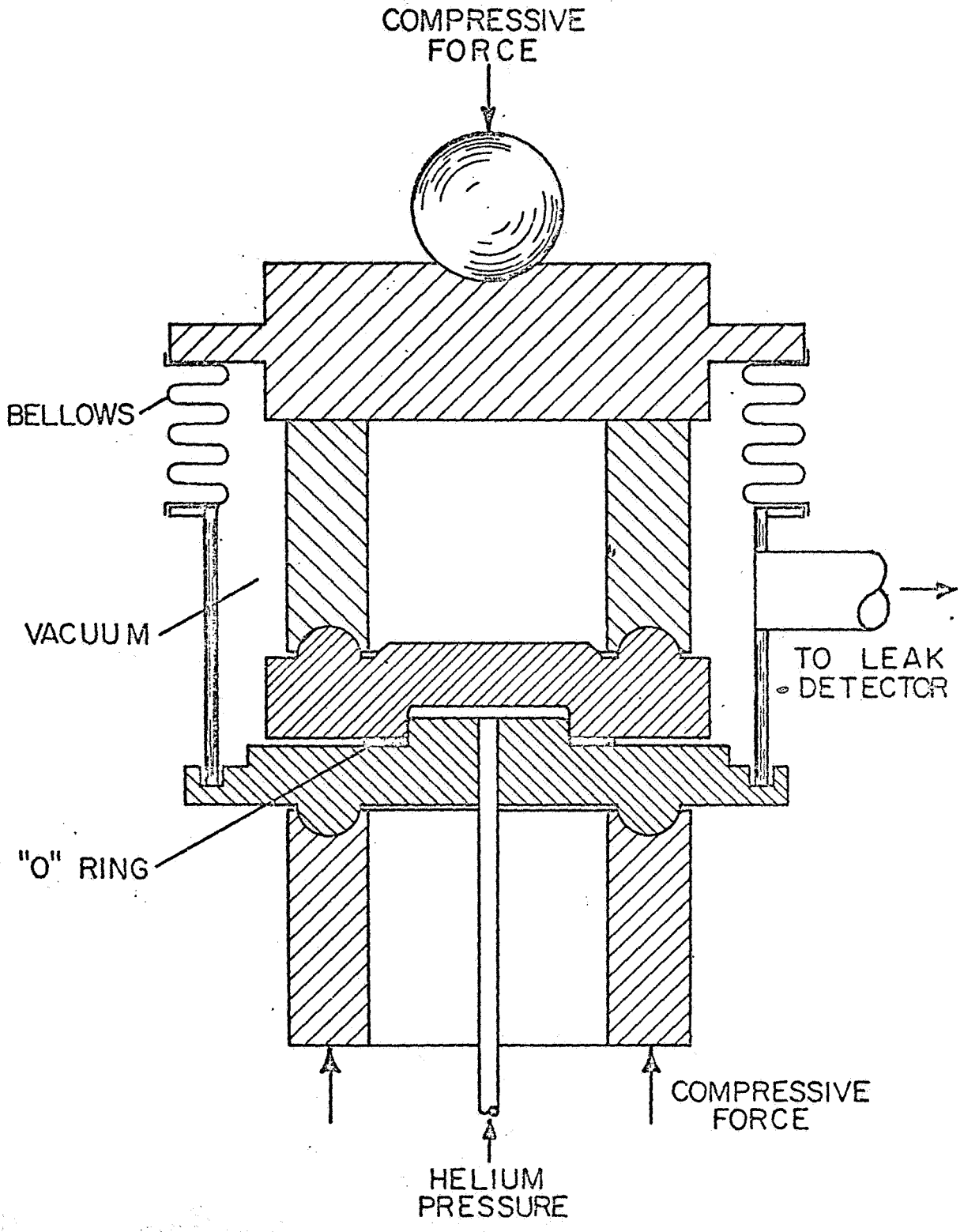



FIG 8. Constant Force Seal Test Apparatus

With this equipment and a selected control group of one inch diameter by 0.140 inch cross section O-rings we determined the minimum force required to maintain tight seals at 76 K with 1000 psig helium on the inside and high vacuum on the outside. The minimum constant force required for the better materials was 5000 to 6000 pounds, which is equivalent to about 1200 pounds net force per linear inch of O-ring, or a net pressure of 4000 to 5000 psi on the compressed O-ring surface. This agreed with results obtained with the force decay apparatus, and led to attempts to lubricate the interfaces with a thin layer of material which would flow plastically at cryogenic temperatures.

To pursue this idea we used a sandwich configuration consisting of two foil or film washers with the O-ring between, as shown in figure 9. Films of polytetrafluoroethylene, lead, aluminum, and indium were tried with neoprene O-rings. Three mil thick indium foil proved to be highly successful when used in this way. Flange loading was reduced by a factor of three, and more reliable seals than ever before were obtained. A net flange load of only 400 pounds per inch of seal circumference was required to separate 1000 psig at 76 K from high vacuum with leakage less than $10^{-6}$ atm cc/sec. Net pressure on the sealing surfaces was reduced to about 1500 psi. It might be mentioned also that the step in the flange was designed for external rather than for internal pressure, as shown in sketch A of figure 10. If the back-up step had been on the outside of the O-ring, as shown in sketch B, the seal could probably have been maintained with even less flange loading.

The indium-elastomer sandwich seals were tested in the constant force apparatus with O-rings of natural rubber, neoprene, polybutadiene, ethyl-acrylate, vinylidene fluoride perfluoropropylene, and chlorosulfonated polyethylene. All performed exceptionally well. Neoprene-indium was also tested in the force decay fixture. While at 76 K this seal was shocked by dropping the heavy fixture on a concrete floor from a height of several inches, a treatment which very few previous seals had sustained without developing leaks. The neoprene-indium seal could not be made to leak by this type of shock treatment.

The elastomer indium sandwich makes effective use of indium's ability to cold flow. A very thin layer of indium remains compressed between the elastomer and the flange surface and continuously fills any openings which tend to form as the surfaces move relative to one another. In this way indium functions as an O-ring lubricant or grease, but one which does not become brittle at cryogenic temperatures. The advantage over plain indium is that there is no continuous cold flow because of frictional

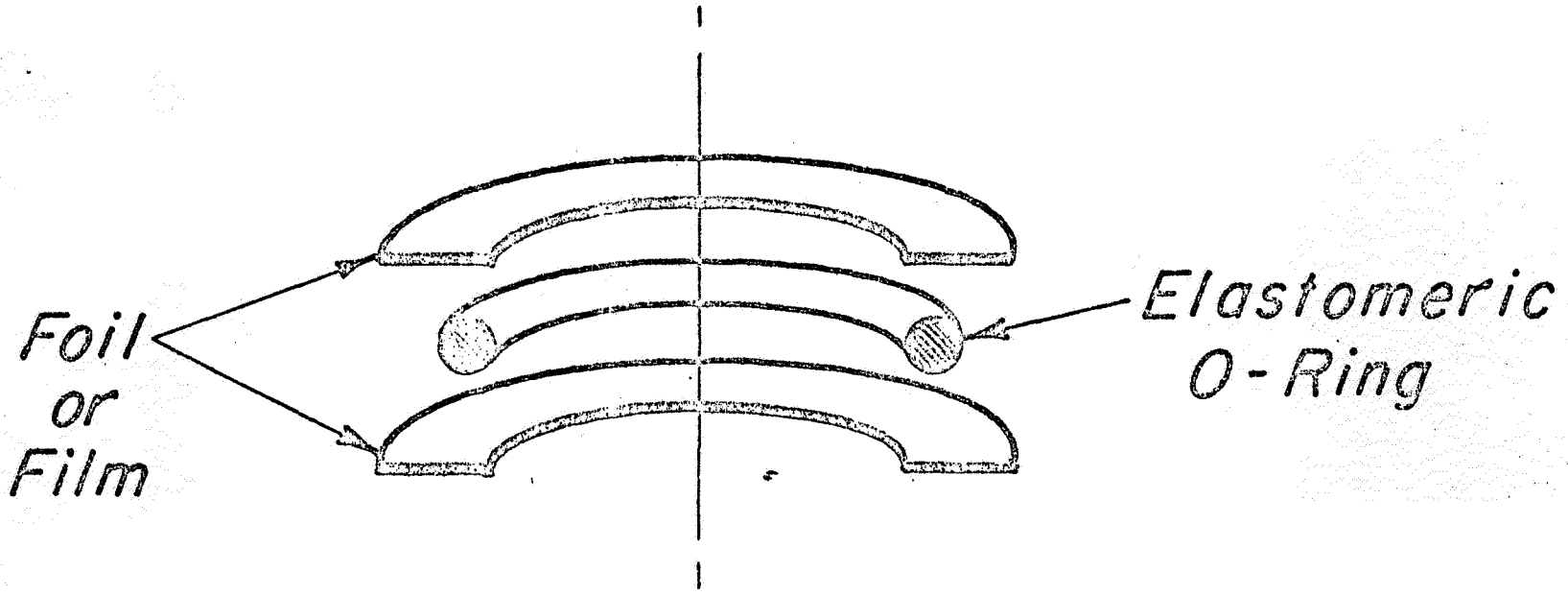
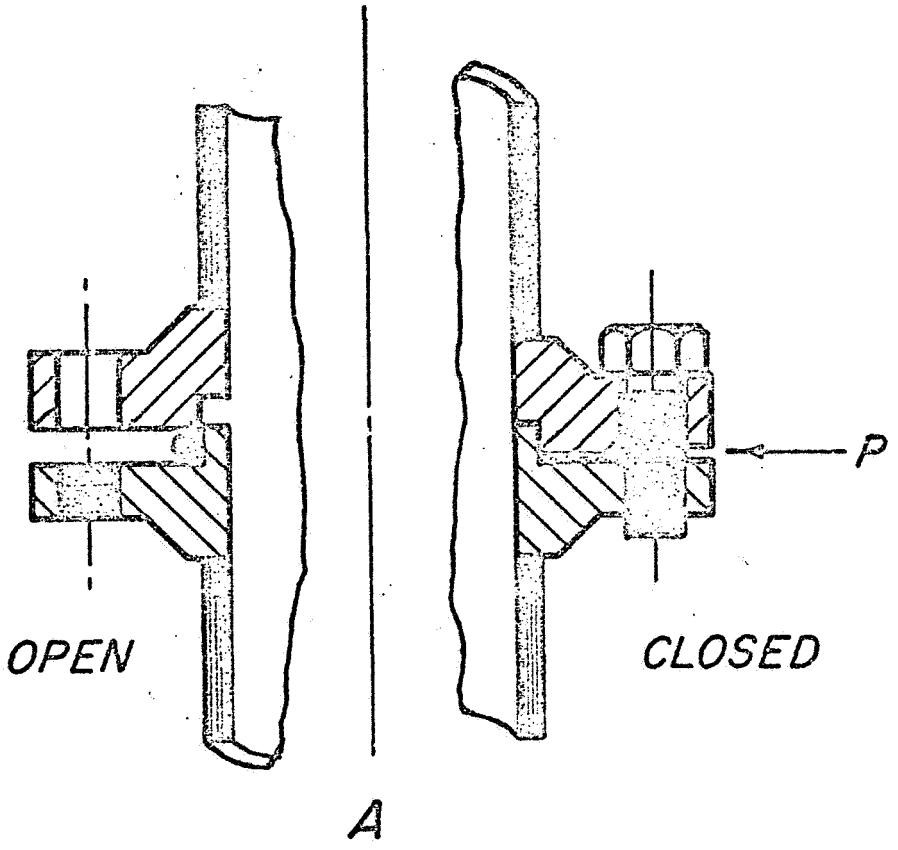



FIG 9. Sandwich Seal Configuration



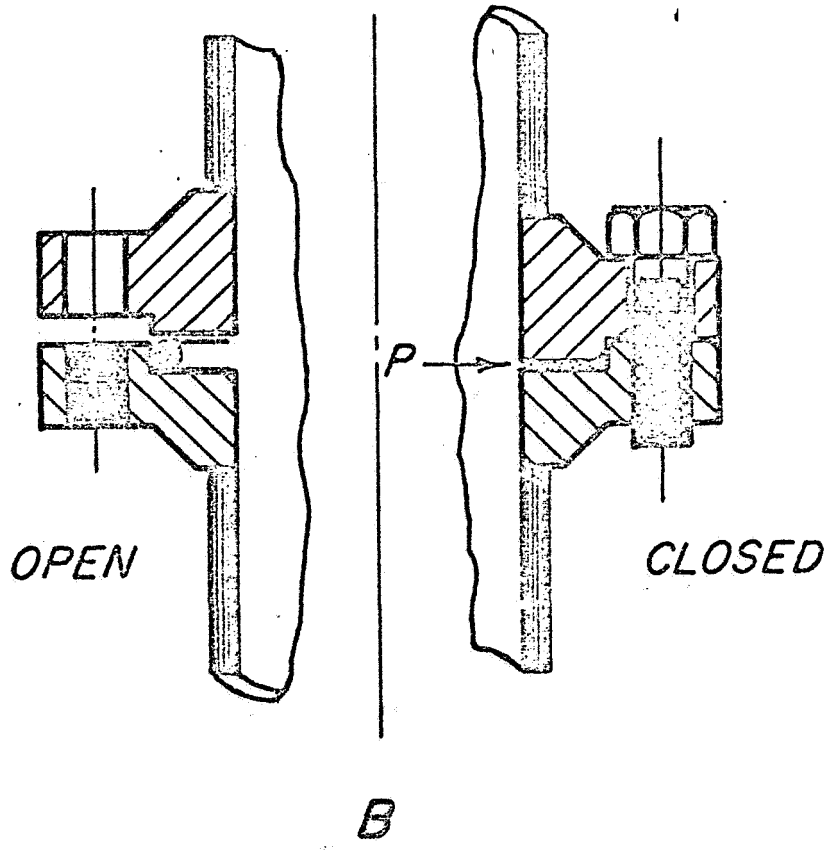




FIG 10. Stepped Flanges

forces on the extremely thin layer of indium which remains between the flange and O-ring. In addition, each time the elastomer warms to its brittle point it comes to life like a compressed spring and reseats the seal. Thus we have a technique for adding elasticity to indium.

With the aid of these two experiments, one showing force decay and seal performance at constant flange separation, the other giving seal performance at constant flange loads, we can restate the principles to be kept in mind for use of static elastomeric seals at cryogenic temperatures. These are:

(1) Choose an elastomer which can be given 80 to 90 percent linear compression without material failure. This requirement is easily met by the elastomers which performed best in the above tests.

(2) Compress at least part of the elastomer cross section 80 to 90 percent, and choose the cross section to give a final compressed thickness no greater than 0.010 inch.

(3) Spring load the compressed elastomer, or use the pressure of the confined fluid, or take advantage of differential thermal contraction (or use two or all of these) to maintain a net sealing pressure of about 5000 psi on the sealing area. This does not require heavy flanges nor high bolt loads because the sealing area can be made small. A seal width of 0.050 inches, which should be ample, will experience a compressive load of 5000 psi with a flange load of only 250 pounds per inch of seal circumference.

(4) If the interface between the elastomer and the flange is lubricated with a thin layer of indium the required net sealing pressure can be reduced to as little as 1500 psi.

## 5. Design Suggestions

We have described a number of experimental flange and elastomer combinations which were developed to test various aspects of the performance of elastomeric O-rings as seals at cryogenic temperatures. Several of these designs were found to make very satisfactory high pressure to high vacuum seals and can be used for many practical low temperature applications.

In conclusion, however, we would like to suggest a few designs which utilize principles which the experimental flanges have helped us to establish. For light weight flanges and low bolt loads one can use:

(1) An O-ring of convenient cross section, but requiring high compression on only a small sealing area.

(2) Spring loading.

(3) Pressure actuation.

(4) Temperature actuation.

For good connector design, one should have:

(1) Simple, symmetrical flanges.

(2) Easy trouble-free assembly.

(3) Solid metal to metal flange contact for transmission of loads through the joint.

Figure 11 shows a stepped flange which can be dimensioned to apply high compression to any desirable fraction of the O-ring cross section. The step can of course be reversed, as shown at B, which is better for vacuum or external pressure, and also holds the O-ring better during assembly of the joint. These stepped designs depend for spring loading on flexing of the flanges before metal-to-metal contact. They are neither pressure nor temperature actuated, and the flanges are not symmetrical, but they are simple and those which we have tested have been reliable.

Figure 12 shows a flange and insert assembly which uses two O-rings in a design analogous to the usual spring loaded and pressure actuated seal. This design has solid metal-to-metal contact for transmitting loads through the joint and does not depend on flange flexing for spring loading. Flanges are simple, symmetrical, and light weight, with bolt loads controlled by the spring constant of the insert beams. This is a good design which combines most of the desirable features which we have enumerated.

Figure 13 shows another method for tucking away part of the cross section of the O-ring and applying a controlled spring load to the remainder, which is used to make the seal. If the beams point out, as shown, the O-ring can be positioned on the O. D. of the insert but the seal is oriented for external rather than internal pressure. For high internal pressure the insert should be reversed as shown in figure 14.

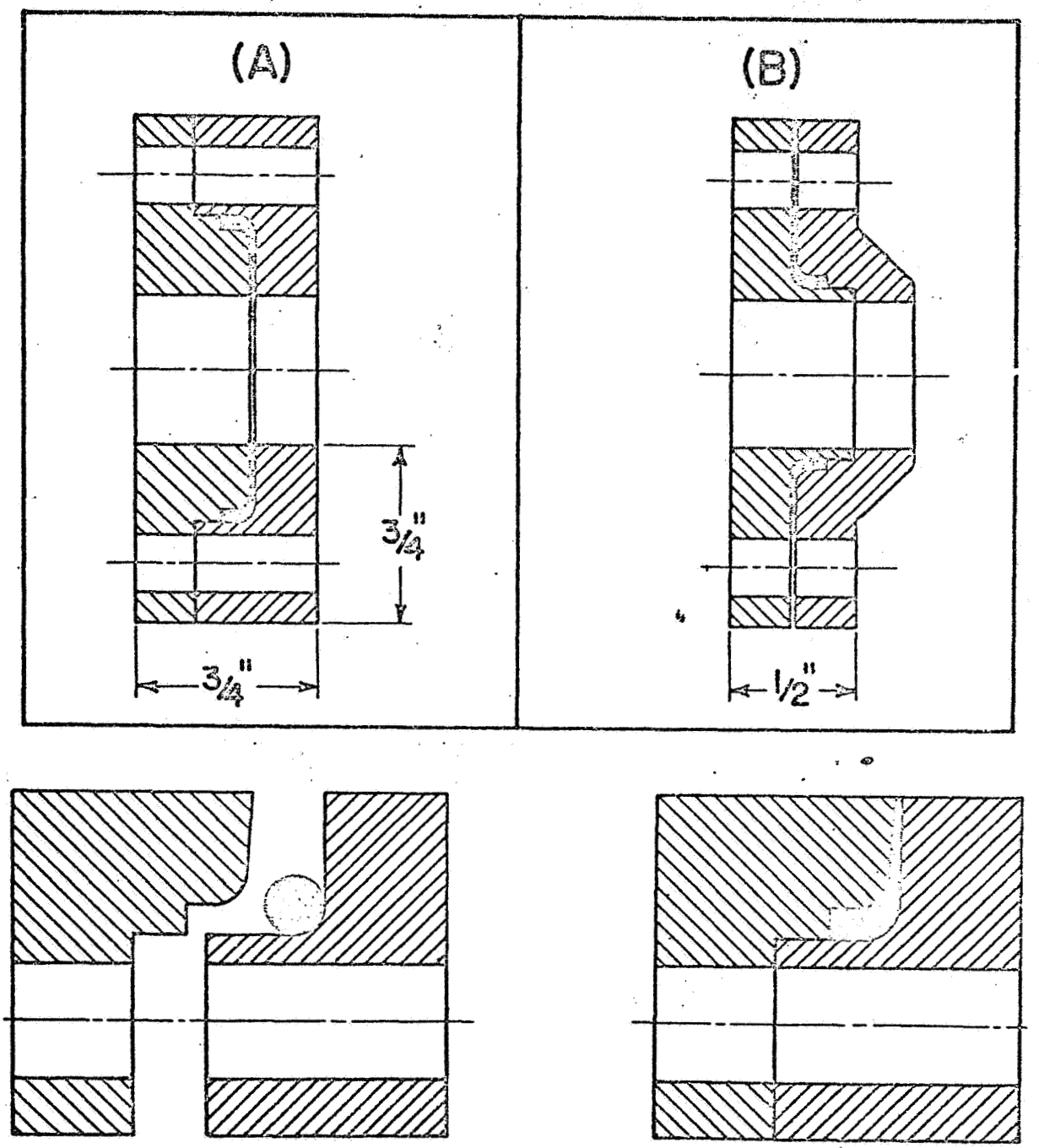





FIG 11. Stepped Flanges With Cross Section Control

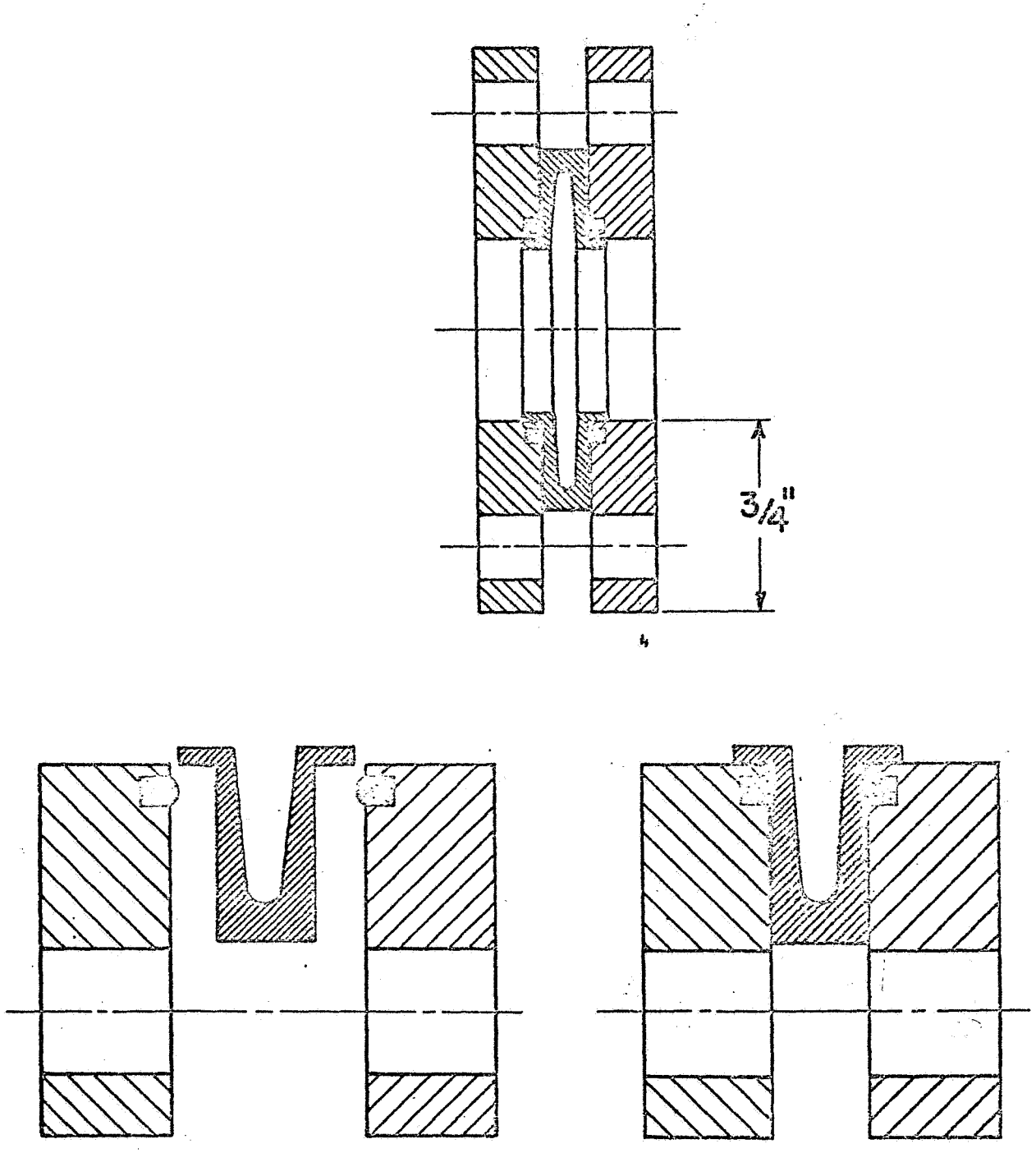



FIG 12. Elastomer Seal, Spring Loaded and Pressure Actuated

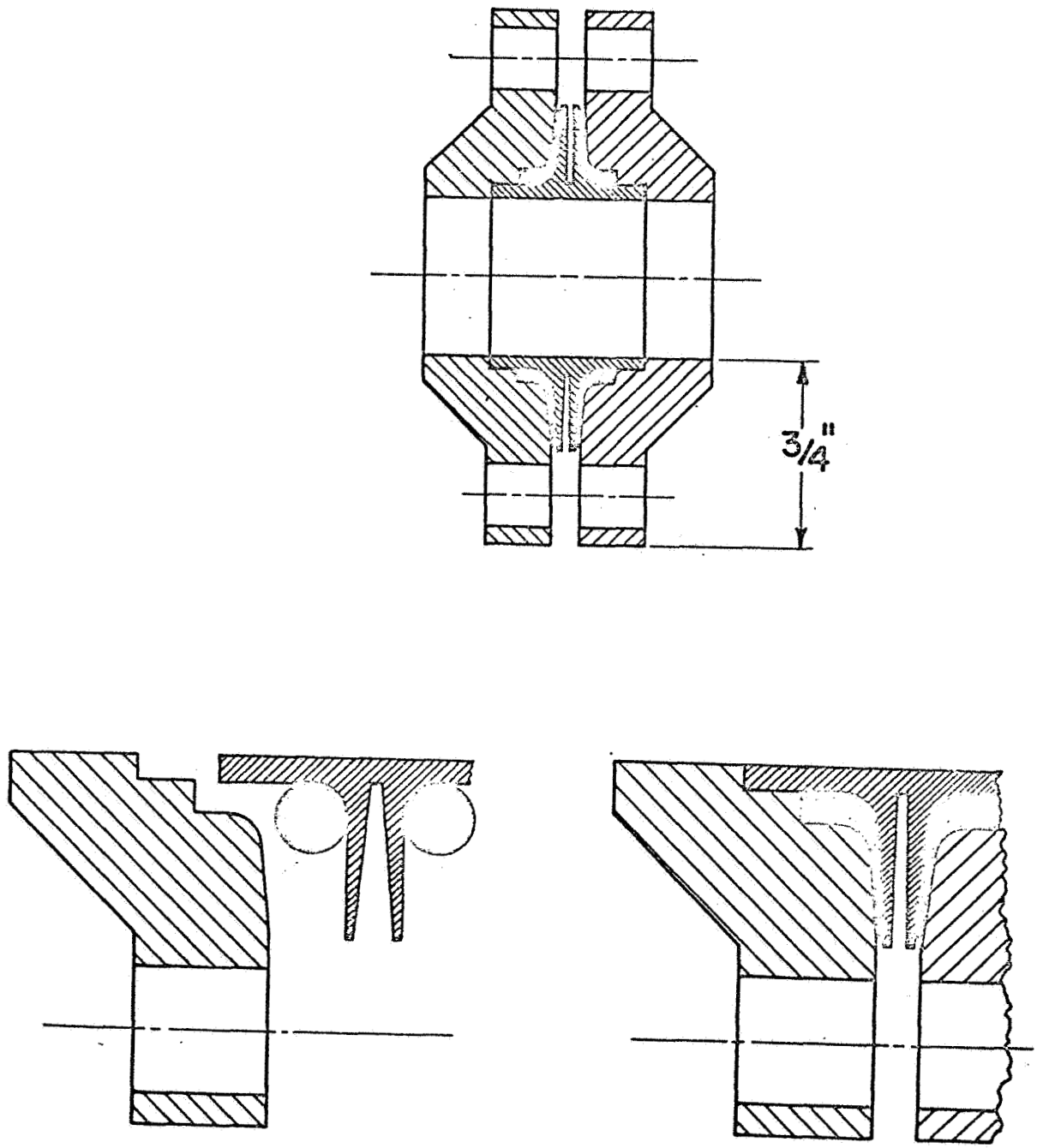



FIG 13. Spring Loaded Seal for External Pressure

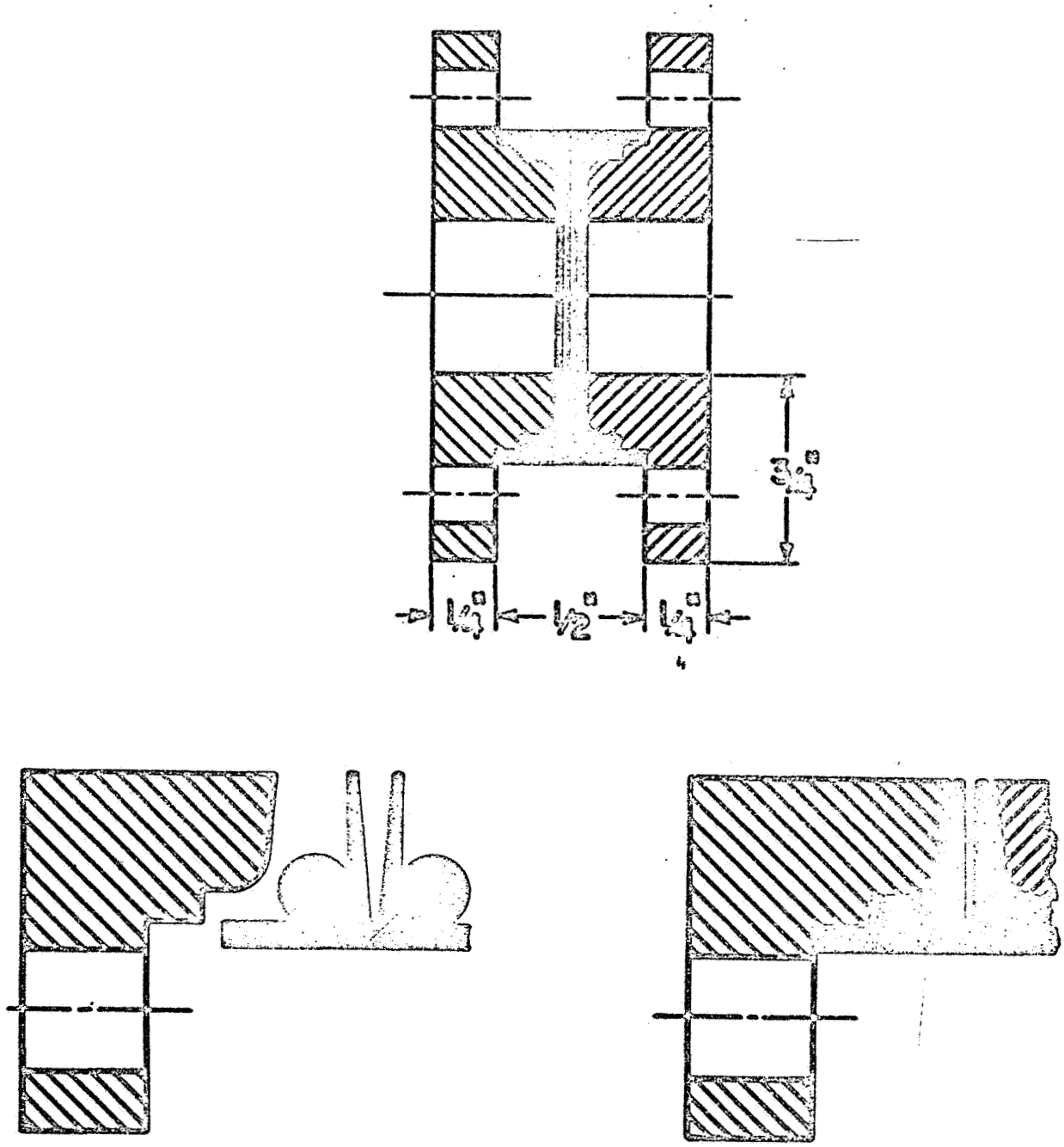


FIG 14. Spring - Loaded Seal for Internal Pressure

A third possibility is shown in figure 15 which spring loads the elastomer through hoop compression of a ring-shaped insert. This design is compact and allows low bolt loads because it utilizes the mechanical advantage of an inclined plane to compress the insert ring and elastomer. It is not suitable for diameters less than about four inches, however, because in these the insert ring may yield in compression before deflecting enough to make room for the elastomer, and subsequently will not have sufficient springback to maintain an effective sealing pressure. *

Materials for these spring loading inserts should be chosen primarily on the basis of elastic properties and yield strength, with thermal expansivity a secondary consideration. If the O-ring is positioned on the O. D. of the insert the seal will tighten during cool-down if the insert shrinks less than the flange. If the O-ring is on the I. D. of the insert, temperature actuation results if the insert shrinks more than the flange.

These and other designs are being studied at the NBS Cryogenic Engineering Laboratory. We are convinced that elastomers should not be overlooked when considering the problem of leak tight separable connectors for service at cryogenic temperatures.

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* Threaded holes between the bolt holes in the flanges are used to disassemble the seal by jacking against the insert skirt.

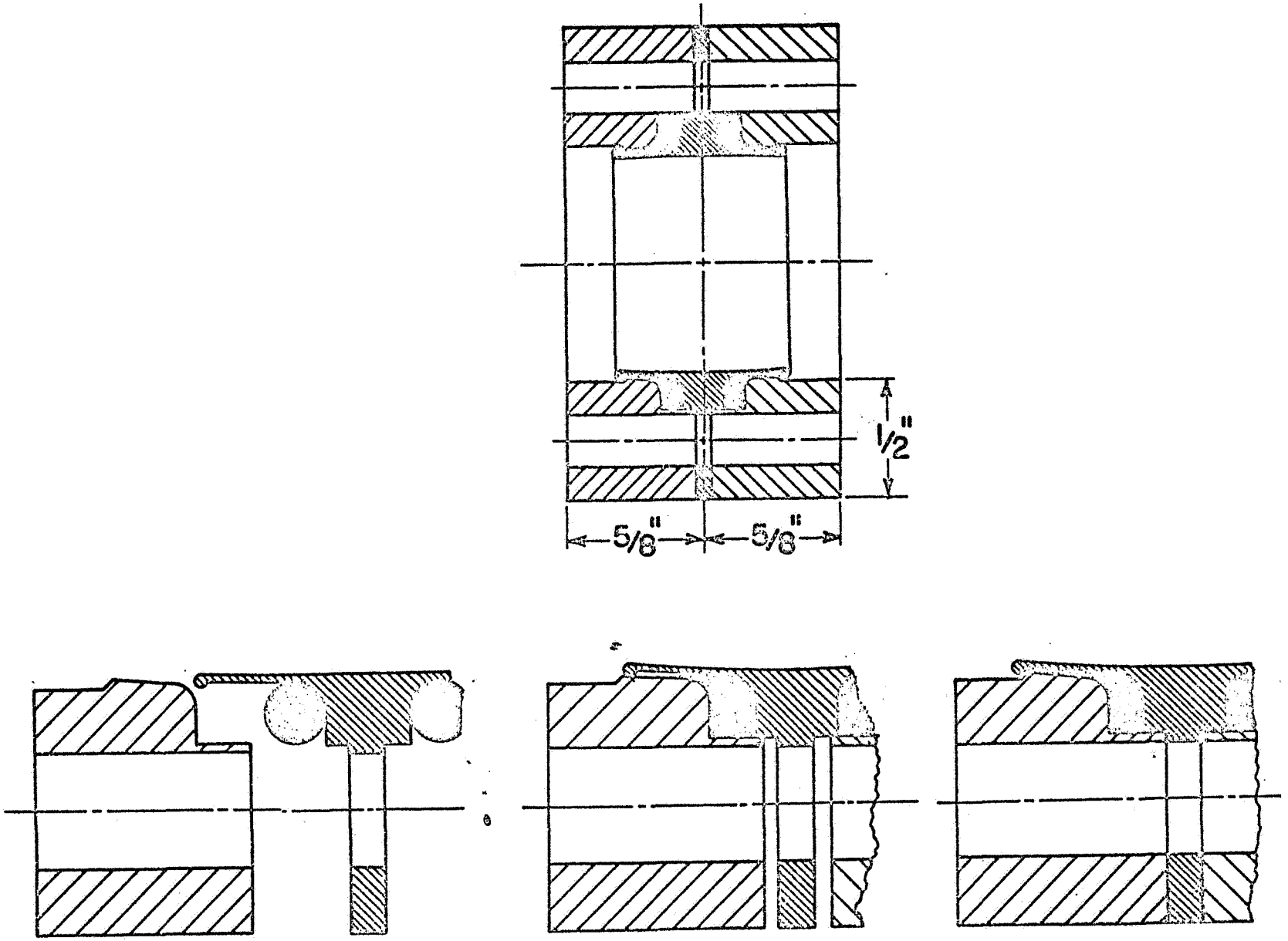


FIG 15. Spring Loaded Seal With Hoop Insert